

CLAIMS

1. A method of producing an interpenetrating polymer network (IPN)
5 comprising the steps of
- i) providing a silicone polymer composition,
- ii) providing one or more monomers for a polymer,
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- iii) providing a solvent for the one or more monomers,
- iv) exposing said silicone polymer composition to said one or more
monomers and said solvent to precipitate monomer within said silicone
15 polymer composition and
- v) polymerizing said monomer to form an IPN,
- wherein said solvent has a surface tension at the exposing step of about 15
20 mN/m or less, such as about 10 mN/m or less, such as about 8 mN/m or
less, such as about 6 mN/m or less, such as about 5 mN/m or less, such as
about 0 mN/m.
2. A method of producing an IPN according to claim 1, wherein said
25 solvent has a surface tension in liquid form of about 15 mN/m or less, such
as about 10 mN/m or less, such as about 8 mN/m or less, such as about 6
mN/m or less, such as about 5 mN/m or less.
3. A method of producing an IPN according to any one of the claims 1 and
30 2, wherein said silicone polymer composition comprises at least 10 %, such
as at least 20 %, such as at least 40 %, such as at least 60 % by weight of
polymer having a backbone consisting of Si and O molecules.
4. A method of producing an IPN according to any one of the claims 1 and
35 2, wherein said silicone polymer composition comprises at least 10 %, such

as at least 20%, such as at least 40%, such as at least 60 % by weight of polymer having a backbone consisting of Si molecules.

5. A method of producing an IPN according to any one of the preceding
5 claims, wherein said silicone polymer composition comprises at least 10 %, such as at least 20 %, such as at least 40 %, such as at least 60 % by weight of polysiloxane polymer.
6. A method of producing an IPN according to any one of the preceding
10 claims, wherein said silicone polymer composition comprises one or more polymers selected from the group consisting of dimethyl polysiloxan, methylphenyl polysiloxane, fluorosilicone rubber, silicone esters, polysiloxanes, polysilanes, chlorosilanes, alkoxysilanes, aminosilanes, polysilanes polydialkylsiloxanes, polysiloxanes containing phenyl
15 substituents, said polymers of the silicone polymer composition optionally being vinyl-functionalized and/or optionally being partially or fully fluorinated.
7. A method of producing an IPN according to any one of the preceding
claims, wherein said silicone polymer composition comprises up to 90 % by
20 weight of non-silicone polymers or co-polymers, fillers and/or additives, said non-silicone polymers e.g. being thermoplastics or thermosets, preferably selected from the group consisting of polyolefins, polyesters, polyurethanes, polycarbonates, polyvinyl polymers, said fillers e.g. being particles or fibres e.g. in the form of minerals or organic fillers, preferably selected from the
25 group consisting of silica, metals, metal oxides, mixed metal oxides, glass beads or glass fibers, and said additives e.g. being selected from the group consisting of adhesion promoters for 2K-constructions, process and plasticizing oils, antioxidants and pigments.
8. A method of producing an IPN according to any one of the preceding
30 claims, wherein said silicone polymer composition is partially vulcanized, such as up to a vulcanization degree (cross-linking degree) of at least 50 %, such as at least 70 %, such as at least 90 % or wholly, such as at least 96 %, such as at least 97 %, such as at least 98 %.

9. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is a vulcanized rubber.

5 10. A method of producing an IPN according to any one of the preceding claims, wherein said one or more monomers are dissolvable in said solvent when said solvent is in liquid state, in gas state or in supercritical state, said monomer preferable being dissolvable in the solvent in its liquid and/or supercritical state.

10 11. A method of producing an IPN according to any one of the preceding claims, wherein said one or more monomers comprise at least 1 % by weight, such as at least 10 % by weight, such as at least 50 % by one or more of the monomers selected from the group consisting of silicone containing monomers such as silanes, such as TEOS (tetraethylorthosilicate
15 or tetraethoxysilane) or chloro- or alkoxy-functional silanes, olefins such as ethylene, propylene, styrene, vinylpyrrolidone, oxygen- and nitrogen-containing monomers such as acrylic derivatives, e.g. acrylic ester and acrylic acid, methacrylic acid and -ester, urethanes, mono- and di-functional alcohols, carboxylic acids, amines, isocyanates, epoxides, aromatic
20 compounds such as aromatics carrying substituents such as alkyl groups and sulfonated aromatics, aromatic resins, imidazol and imidazol derivatives, pyrazoles, quaternary ammonium compounds, polyurethane prepolymers and epoxy resins.

25 12. A method of producing an IPN according to any one of the preceding claims 1-10, wherein said one or more monomers comprise at least 50 % by weight of vinylpyrrolidone.

30 13. A method of producing an IPN according to any one of the preceding claims 1-10, wherein said one or more monomers comprise at least 50 % by weight of olefins.

35 14. A method of producing an IPN according to any one of the preceding claims 1-10, wherein said one or more monomers comprise at least 50 % by weight of silanes.

15. A method of producing an IPN according to any one of the preceding claims, wherein said one or more monomers are free radical polymerizable.

5 16. A method of producing an IPN according to any one of the preceding claims, wherein the solvent is one or more hydrocarbons or carbon-containing compounds or a composition comprising hydrocarbons or carbon-containing compounds with a Hildebrand solubility of below 11, preferably 8 or less, the silicone polymer composition preferably being exposed to the
10 solvent in its supercritical state or near its supercritical state, where the term 'near its supercritical state' means that the total pressure is at least 10 bar.

17. A method of producing an IPN according to any one of the preceding claims, wherein the solvent comprises one or more compounds from the
15 group of C₁-C₁₂ hydrocarbons or carbon-containing compounds, preferably C₁-C₄ hydrocarbons, more preferably selected from the group consisting of methane, ethane, propane, propene, isobutane, butane, butene, isobutene, methanol, acetone, N₂O and CO₂, more preferably the solvent comprises CO₂, the silicone polymer composition being exposed to the solvent in
20 supercritical state.

18. A method of producing an IPN according to any one of the preceding claims, wherein the solvent has a surface tension in liquid form of about 15 mN/m or less, such as about 10 mN/m or less, such as about 8 mN/m or
25 less, such as about 6 mN/m or less, such as about 5 mN/m or less, the silicone polymer composition being exposed to the solvent while it is in one or more of its states liquid state, gas state and supercritical state.

19. A method of producing an IPN according to claim 18, wherein said
30 solvent comprises at least 50 %, such as at least 75 %, such as at least 90 % by weight of one or more of the components selected from the group consisting of CO₂, and N₂O, and C₁-C₅ hydrocarbons, the solvent preferably comprising at least 50 %, such as at least 90 % of CO₂.

20. A method of producing an IPN according to any one of the preceding claims, wherein the solvent comprises a surfactant preferably selected from the group of anionic, cationic, non-ionic and amphoteric surfactants, said solvent preferably comprising up to 5 % by weight, such as between 0.001-
5 50 grams of surfactant per kg solvent.

21. A method of producing an IPN according to any one of the preceding claims, wherein a radical starter is incorporated into said silicone polymer, preferably by physical compounding, by swelling or impregnation in dissolved
10 condition, or by co-impregnation with the one or more monomers, the amount of radical starter preferably being sufficient to initiate the polymerization.

22. A method of producing an IPN according to any one of the preceding
15 claims, wherein said one or more monomers are dissolved in the solvent together with a radical starter, the amount of radical starter preferably being sufficient to initiate the polymerization.

23. A method of producing an IPN according to any one of the claims 21
20 and 22, wherein the amount of radical starter is at least 0.01 mol % of the monomer, such as at least 0.01 mol % of the monomer, such as at least 0.1 mol % of the monomer, such as at least 0.5 mol % of the monomer, such as at least 1 mol % of the monomer, such as between 1 and 10 mol % of the monomer.

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24. A method of producing an IPN according to any one of the preceding claims 21-23, wherein said radical starter is selected from the group consisting of peroxides such as dicumylperoxide or azo-compounds such as azo-bis-iso-butyronitril, or photo-labile compounds such as benzoyl-based
30 radical starters, said radical starter preferably being heat- or radiation activatable.

25. A method of producing an IPN according to any one of the preceding claims, wherein said monomer is dissolved in liquid solvent, said silicone

polymer composition being exposed to said solvent while the solvent is in liquid state, in gas state and/or in supercritical state.

26. A method of producing an IPN according to any one of the preceding
5 claims 1-24, wherein said monomer is dissolved in gas solvent, said silicone polymer composition being exposed to said solvent while the solvent is in liquid state, in gas state and/or in supercritical state.

27. A method of producing an IPN according to any one of the preceding
10 claims 1-24, wherein said monomer is dissolved in supercritical solvent, said silicone polymer composition being exposed to said solvent while the solvent is in liquid state, in gas state and/or in supercritical state.

28. A method of producing an IPN according to any one of the preceding
15 claims, wherein said silicone polymer composition is exposed to said solvent for a sufficient time to swell the silicone polymer composition with the solvent, preferably to swell the silicone polymer composition with at least 0.01 %, such as at least 0.1 %, such as at least 1 % by weight of solvent calculated on the weight of the silicone polymer composition.

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29. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is exposed to said solvent under varying pressure, the pressure preferably being decreased after at least part of the solvent has been swelled into the silicone polymer
25 composition whereby monomer(s) precipitate within the silicone composition.

30. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is exposed to said solvent under varying temperature, the temperature preferably being decreased after
30 at least part of the solvent has been swelled into the silicone polymer composition whereby monomer(s) precipitate within the silicone composition.

31. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is exposed to said solvent
35 under conditions where the solvent is in a first state, followed by a change of

conditions whereby the solvent changes to a second state, said first state preferably being liquid state or supercritical state and said second state preferably being gas state.

5 32. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is exposed to said solvent with said one or more monomers for a sufficient time to precipitate at least 0.01 %, such as at least 0.1 %, such as at least 1 % by, such as up to about 50 % by weight of monomer(s) of the total weight of the silicone polymer
10 composition.

33. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is exposed in two or more steps to said solvent with one or more monomers to provide at total
15 monomer precipitation of at least 0.01 %, such as at least 0,1 %, such as at least 1 % by, such as up to about 50 % by weight of monomer(s) of the total weight of the silicone polymer composition, said two or more steps being equal or different from each other e.g. with respect to solvent, monomer amounts and/or exposing time.

20 34. A method of producing an IPN according to any one of the preceding claims 31 and 32, wherein said silicone polymer composition is exposed to said solvent with said one or more monomers for a total time of at least 1 min, such as for 5 min, such as for 20 min or more.

25 35. A method of producing an IPN according to any one of the preceding claims 1-16, wherein said silicone polymer composition is treated with the solvent in or near its supercritical state (for extracting residues) followed by feeding of the monomer into the reaction chamber where the monomer is
30 dissolved in the solvent in or near its supercritical state while the silicone polymer composition simultaneously is exposed to the solvent.

36. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition is exposed to a solvent
35 consisting essentially of carbon dioxide and carrying the monomer(s) in or

near its supercritical state, for a sufficient time to swell the silicone polymer composition with the solvent with monomer(s).

5 37. A method of producing an IPN according to any one of the preceding claims, wherein said silicone polymer composition prior to the exposing step is shaped, preferably by extrusion, injecting-moulding, calendaring, pressing or cutting.

10 38. A method of producing a polymeric unit (for automotive or telecom use) comprising the steps of:

-producing an IPN unit in the desired shape according to any one of the preceding claims 1-37, and

15 -treating the surface thereof, e.g. by plasma or flame surface activation and subsequent painting, or immediate painting or coating.

20 39. A method of producing a polymeric unit according to claim 38 wherein the silicone polymer composition used for the IPN is transparent.

40. A method of producing a polymer unit according to any one of the claims 38 and 39, wherein the one or more monomers comprise one or more from the group of styrene, acrylics and urethanes

25 41. A method of producing a polymer membrane comprising the steps of:

-producing an IPN unit shaped as a membrane according to any one of the preceding claims 1-37, wherein the membrane has a thickness between 5 and 1000 μm .

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42. A method of producing a polymer membrane according to claim 41 wherein the amount of monomer precipitated into the silicone polymer composition is 5 % by weight or less, such as 2% or less or such as 1% and less.

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43. A method of producing a polymer membrane according to any one of the claims 41 and 42, wherein the membrane is a membrane for separation of gases or liquids.

5 44. A method of producing a polymer membrane according to any one of the claims 41 and 42, wherein the membrane is a fuel cell membrane, said membrane preferably being less permeable to gases than the original
10 silicone composition at least by a factor of 2, and whereby the conductivity for protons (H⁺) preferably is increased in comparison with the original silicone composition by incorporation of proton-conducting monomers such as imidazol or imidazol derivatives, the conductivity for protons (H⁺) preferably being increased in comparison with the original silicone composition by a factor of at least 10, such as at least 100.

15 45. An IPN obtainable according to any one of the preceding claims 1-37.

46. An IPN according to claim 45 wherein the IPN is a full IPN comprising at least two interpenetrating, individually cross linked networks.

20 47. An IPN according to any one of the preceding claims 45 and 46 wherein the IPN is essentially free of organic solvents.

48. An IPN according to any one of the preceding claims 45-47 wherein the IPN comprises a network of a polymer selected from the group consisting of
25 polyvinylpyrrolidone.

49. An automotive part obtainable according to any one of the claims 38-40.

50. A telecom part obtainable according to any one of the claims 38-40.
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51. A medical device such as catheter, part of a pace maker and an implant.

52. A gas separation membrane obtainable according to any one of the claims 41-43.
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53. A fuel cell membrane obtainable according to any one of the claims 41, 42 and 44.

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